

Fabrication and characterization of iron oxide nanoparticles reinforced vinyl-ester resin nanocomposites

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Abstract

Robust magnetic vinyl ester resin nanocomposites reinforced with iron oxide (Fe_2O_3) nanoparticles were fabricated. The particle functionalization with a bi-functional coupling agent methacryloxypropyl-trimethoxysilane (MPS) was observed to have a significant effect on the curing process and subsequent physical properties of the nanocomposites. Particle functionalization favors the composite fabrication with a lower curing temperature as compared to the as-received nanoparticles filled vinyl ester resin nanocomposites. Thermogravimetric analysis showed an increased thermo-stability in the functionalized nanoparticles filled vinyl ester resin nanocomposites as compared to the unmodified nanoparticle filled counterparts. The more uniform particle dispersion and the chemical bonding between nanoparticle and vinyl ester resin matrix were found to contribute to the increased thermal stability and enhanced tensile strength. The nanoparticles become magnetically harder after incorporation into the vinyl ester resin matrix.

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1. Introduction

Nanomaterials dramatically different from their bulk or atomic counterparts have attracted much interest due to their unique physicochemical properties [1] for a wide range of potential device applications such as UV lasers [2], solar cells [3,4], high-sensitivity chemical gas [5] or volatile organic compound [6] sensors, and DNA sequence [7] sensors. Nanostructural materials such as nanoparticles (NPs) or nanofibers have been used as fillers in both the polymeric nanocomposites [8,9] to improve the mechanical, electric, electronic and optical properties, and the metallic nanocomposite [10] to control the electrodeposition. Polymer nanocomposites reinforced with

inorganic nanoparticles have attracted much interest due to their lightness, homogeneity, cost-effective processability, and tunable physical properties [9,11–13]. Vinyl-ester resin, as a structural polymer, was chosen as a polymer matrix in current study due to the fact that the cured resins are thermosetting with a network structure possessing high resistance to the moisture and chemicals, and good mechanical properties. Thus the resultant composites have the potential applications in fabrication and building materials such as electrodeposition tanks, automotive parts and marine vessels which require superior mechanical properties and/or high resistance to harsh environments such as strong acid or base. Furthermore, the functional groups of the polymer surrounding the nanoparticles enable these nanocomposites as good candidates for various applications such as site-specific molecule targeting in biomedical areas [14].

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The existing challenges in the composite fabrication are to obtain uniform filler dispersion and to introduce strong chemical bonding between the nanoparticles and the polymer matrix, which are necessary to provide a high tensile strength due to local stress within the nanocomposite. The interfacial interactions between fillers and polymer matrix play a crucial role in determining the quality and properties of the nanocomposites [15]. The poor bonding linkage between the fillers and the polymer matrix such as the composites made by simple mixing [15] will introduce artificial defects, which consequently result in a deleterious effect on the mechanical properties of the nanocomposites [16]. Introducing good linkages between the fillers and the polymer matrix is still a challenge for specific composite fabrication. However, appropriate chemical engineering treatment of the nanofiller surface by introducing proper functional groups could improve both the strength and toughness of the subsequent composites with improved compatibility between the nanofillers and the polymer matrix, and make the nanocomposites stable in harsh environments as well [17]. Thus, surface functionalization of nanoparticles with a surfactant or a coupling agent is important not only to stabilize the nanoparticles [18] during processing but also to render them compatible with the polymer matrix.

In this paper, the effect of particle functionalization by a bi-functional methacryloxypropyl-trimethoxysilane (MPS) on the vinyl ester resin curing process and the optimum conditions for high-quality nanocomposite fabrication were investigated. The functionalized iron oxide nanoparticles reinforced vinyl ester nanocomposites showed enhanced mechanical properties under tensile study. The nanocomposites containing the functionalized iron oxide nanoparticles showed improved thermal stability as compared with the nanocomposites reinforced with the as-received iron oxide nanoparticles. The nanoparticles were found to be magnetically harder (larger coercivity) after the nanoparticles were dispersed in the vinyl ester resin matrix.

2. Experimental

2.1. Materials

The polymeric matrix used was a vinyl ester resin, Derakane momentum 411–350 (manufactured by the Dow Chemical Company), which is a mixture of 55 wt% vinyl ester with an average molecule weight of 970 g/mol and 45 wt% styrene monomers. Styrene with only one unsaturated carbon–carbon double bond provides linear chain extension. Vinyl-ester monomers with two reactive vinyl end groups enable the cross-linking for network formation. The liquid resin has a density of 1.045 g/cm³ and a viscosity of 350 centipoises (cps) at room temperature. Iron oxide (γ -Fe₂O₃, Nanophase Technologies) nanoparticles with an average diameter of 23 nm and a specific surface area of 45 m²/g were functionalized and used as nanofillers for the nanocomposite fabrication. Trigonox 239-A (curing

catalyst or initiator, organic peroxide, liquid) was purchased from Akzo Nobel Chemicals. Cobalt naphthenate (CoNap, OM Group, Inc.) was used as a catalyst promoter (accelerator) to decompose the catalyst at room temperature. Methacryloxypropyl-trimethoxysilane (MPS) and tetrahydrofuran (THF, anhydrous) were purchased from Sigma–Aldrich Chemical Company. All the chemicals were used as-received without further treatment.

2.2. Surface functionalization of iron oxide nanoparticles

The nanoparticle functionalization follows procedures similar to our earlier reported nanoparticle process [19–21] and is described as follows. Fe₂O₃ nanoparticles (13.38 g, 83.8 mmol) were added into a mixture of 4 g MPS and 30 ml THF. The resulting colloidal suspension was ultrasonically stirred (Branson 1510) for 1 h and precipitated by a permanent magnet at room temperature. The precipitated nanoparticles were rinsed with THF to remove excessive MPS for subsequent nanocomposite fabrication and the dried nanoparticles were used for further particle characterization.

2.3. Nanocomposite fabrication

The as-received Fe₂O₃ nanoparticles or MPS functionalized Fe₂O₃ nanoparticles were dispersed into 30 ml resin on a specific weight percentage basis. The dispersion was carried out in an ice-water ultrasonic bath for about 1 h. The above particle-suspended solution was then ultrasonically stirred in an ice-water ultrasonic bath until the temperature was cooled down. Then, 2.0 wt% catalyst (initiator) was added into the nanoparticle/resin solution, which was stirred and degassed for 2 min. Next, 0.3 wt% promoter was added and mixed quickly. The mixed viscous solution was poured into silicone rubber molds. The curing via free-radical bulk copolymerization or homopolymerization initiated by the catalyst was done at 85 °C for 1 h under normal atmospheric conditions and cooled down to room temperature naturally in the oven following procedures similar to those used for our reported alumina nanoparticles filled vinyl-ester resin nanocomposite fabrication [19]. A viscous liquid solution was still observed in the mold after this curing process, indicating that nanofilling materials have a significant effect on the quality of fabricated nanocomposites. However, high-quality nanocomposites were formed by room temperature curing for 24 h followed by postcuring at 100 °C for 2 h.

2.4. Characterization

A Fourier transform infrared (FT-IR) spectrometer was used to test the physicochemical interaction between MPS and Fe₂O₃ nanoparticles, and the change of the MPS functional groups after the nanoparticle modification. FT-IR spectra were recorded in a FT-IR spectrometer (Jasco, FT-IR 420) in transmission mode under dried nitrogen

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